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Hitachi S-570 Scanning Electron Microscope with Energy-Dispersive X-Ray Spectroscopy

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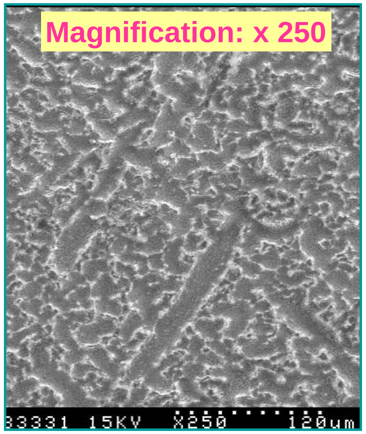
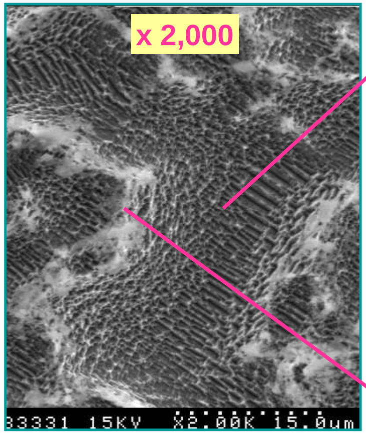
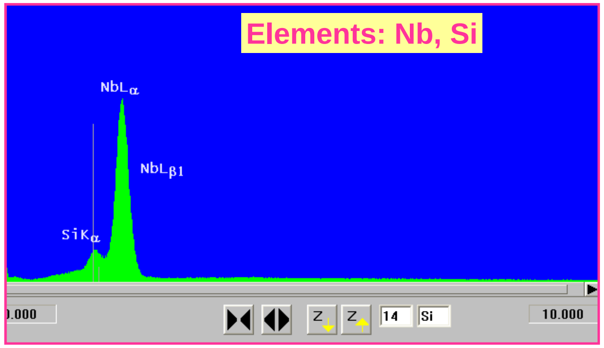
The **scanning electron microscope** (**SEM**) is a type of electron microscope capable of producing high-resolution images of the sample surface. The high energy beam of electrons scans the surface of a sample. From the interaction of the incident electrons with the sample's surface, different types of signals are gathered: secondary electrons, characteristic x-rays, and back scattered electrons. The most common imaging mode monitors low energy (<50 eV) secondary electrons. Due to their low energy, these electrons originate within a few nanometers from the surface. The electrons are detected by an scintillator-photomultiplier device and the resulting signal is rendered into a two-dimensional intensity distribution that can be viewed and saved as a Digital image. Using this technique, resolutions less than 200 nm are possible.

Digital image 1.



Digital image 2.

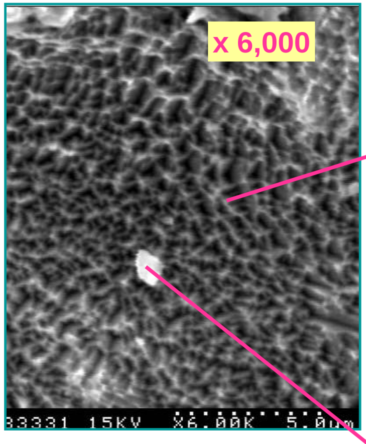




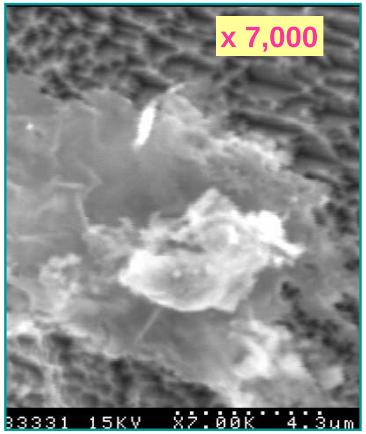
Energy dispersive X-ray spectroscopy (EDS or EDX) is an analytical technique used for the chemical characterization of a surface. The x-ray released in interaction between the high-energy electron beam and the surface is detected and analyzed by the energy dispersive spectrometer. In the specrtometer, X-ray energy is converted into voltage signals; this information is sent to a pulse processor, which measures the signals and passes them onto an analyzer for data display and analysis. EDX data is often plotted as x-ray energy vs. count rate. The peaks correspond to characteristic elemental emissions that are a consequence of specific electron configuration of atom. The software is used for qualitative and quantitative analysis of samples' surface composition.

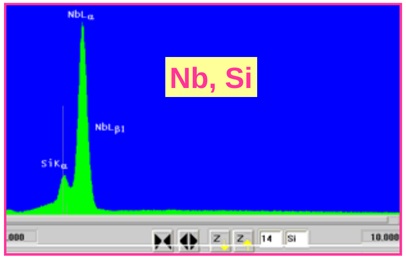
Magnification increases

"Trench"

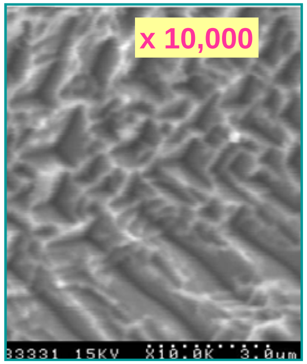


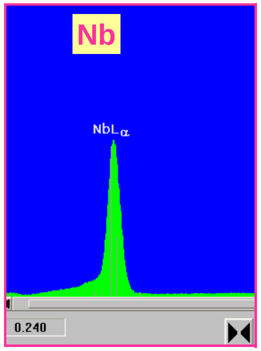
"Ridge'



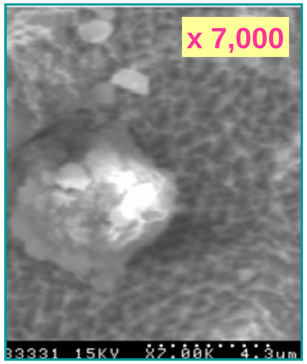


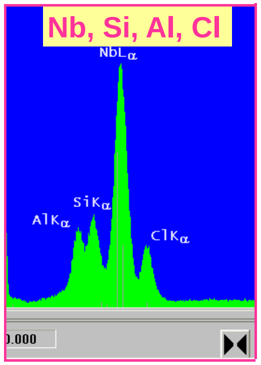
Niobium surface





Particle





SEM/EDX Application Example:

Bulk Nb surface was examined after exposure to microwave glow discharge in Ar/Cl_2 reactive gas. The typical rough surface, with characteristic features that resemble "trenches" and "ridges", is shown on Digital image 1. and 2. EDX analysis is showing presence of traces of silicon that was not expected. Focusing on the "ridges" showed that almost all Si is concentrated on the top of them. Possible source of Si is sanding paper used to mechanically polish samples before exposure to discharge. Etching rate of the silicon in Cl_2 gas is relatively smaller comparing to Nb etching rate. Therefore accumulation of Si prevents etching of Nb and increase roughness of sample surface. To obtain smooth a surface special attention must be devoted to preparation of surface before exposure to discharge. EDX of "trenches" reveals pure Nb with characteristic "facets" corresponding to crystalline orientation of Nb grain. Additional impurities in the shape of particles attached to energy rich centers can also be noticed. Size of imaged particle is ~ 4 μm. Additional Ar discharge with strong gas flow can be applied to Nb sample surface to remove this type of impurities in the final step of plasma etching.